

PCT

WORLD INTELLECTUAL PROPERTY ORGANIZATION
International Bureau



INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 5 :

C11D 1/02, 1/72, 3/50

A1

(11) International Publication Number:

WO 94/26856

(43) International Publication Date: 24 November 1994 (24.11.94)

(21) International Application Number:

PCT/US94/04329

(22) International Filing Date:

20 April 1994 (20.04.94)

(30) Priority Data:

93201287.5

6 May 1993 (06.05.93)

EP

(34) Countries for which the regional or
international application was filed:

BE et al.

(81) Designated States: AU, CA, JP, US.

Published

With international search report.

(71) Applicant (for all designated States except US): THE PROCTER & GAMBLE COMPANY [US/US]; One Procter & Gamble Plaza, Cincinnati, OH 45202 (US).

(72) Inventors; and

(75) Inventors/Applicants (for US only): BAZZOLI, Gianni [IT/BE]; Rue Leis 26, B-1040 Brussels (BE). RICCI, Carlo [IT/IT]; Via Dante de Blasi, 70, I-00151 Roma (IT).

(74) Agents: REED, T., David et al.; The Procter & Gamble Company, 5299 Spring Grove Avenue, Cincinnati, OH 45202 (US).

(54) Title: MIXING ORDER TO PREPARE AQUEOUS DETERGENT COMPOSITIONS

(57) Abstract

A process is disclosed for the manufacture of an aqueous detergent composition comprising a substantial amount of organic components. By incorporating said organic components in a specific order, a finished product is obtained which is clear and transparent.

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AT	Austria	GB	United Kingdom	MR	Mauritania
AU	Australia	GE	Georgia	MW	Malawi
BB	Barbados	GN	Guinea	NE	Niger
BE	Belgium	GR	Greece	NL	Netherlands
BF	Burkina Faso	HU	Hungary	NO	Norway
BG	Bulgaria	IE	Ireland	NZ	New Zealand
BJ	Benin	IT	Italy	PL	Poland
BR	Brazil	JP	Japan	PT	Portugal
BY	Belarus	KE	Kenya	RO	Romania
CA	Canada	KG	Kyrgyzstan	RU	Russian Federation
CF	Central African Republic	KP	Democratic People's Republic of Korea	SD	Sudan
CG	Congo	KR	Republic of Korea	SE	Sweden
CH	Switzerland	KZ	Kazakhstan	SI	Slovenia
CI	Côte d'Ivoire	LI	Liechtenstein	SK	Slovakia
CM	Cameroon	LK	Sri Lanka	SN	Senegal
CN	China	LU	Luxembourg	TD	Chad
CS	Czechoslovakia	LV	Latvia	TG	Togo
CZ	Czech Republic	MC	Monaco	TJ	Tajikistan
DE	Germany	MD	Republic of Moldova	TT	Trinidad and Tobago
DK	Denmark	MG	Madagascar	UA	Ukraine
ES	Spain	ML	Mali	US	United States of America
FI	Finland	MN	Mongolia	UZ	Uzbekistan
FR	France			VN	Viet Nam
GA	Gabon				

5

10

MIXING ORDER TO PREPARE AQUEOUS**DETERGENT COMPOSITIONS**

15

20

25

Technical field

The present invention relates to a process for the manufacture of an aqueous detergent composition. The present invention relates more particularly to a mixing order to prepare such a composition comprising a substantial amount of organic components.

35

Background of the invention

Aqueous, detergent compositions have been described extensively in the art. More specifically it is well known that such compositions may be formulated as solutions comprising water and organic components such as surfactants, perfumes, stabilizers and the like. These organic components are predominantly poorly soluble or even insoluble in water. Therefore, there is a natural tendency for said compositions to be unclear, i.e. turbid, as said organic components may not be

40

fully dissolved in the aqueous matrix. This becomes even more of a problem as the total proportion of organic components is increased. This phenomenon results in mitigated consumer acceptance. Therefore, the problem the present invention seeks to solve is to avoid turbidity in the compositions finally obtained.

In response to this technical problem, applicants have found that by incorporating the different organic components in a specific order during the manufacture of such an aqueous detergent composition, a clear and transparent final composition could be obtained.

It is thus an object of the present invention to obtain an aqueous detergent composition which is clear and transparent.

Applicant's European patent application EP-A-517 996 discloses stable hydrogen peroxide-containing bleaching compositions which are made by mixing together all ingredients except for hydrogen peroxide which is added as a final step in the process. Said application discloses nothing about the aforementioned turbidity problem.

Summary of the invention

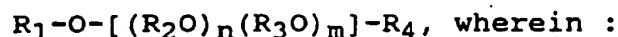
The present invention includes a process for the manufacture of an aqueous detergent composition comprising a nonionic surfactant, an anionic surfactant and a perfume. Said process includes the steps of :

- mixing said nonionic surfactant in said water (composition A);
- dissolving said perfume in said anionic surfactant (composition B);
- thereafter mixing said composition B in said composition A (composition C).

Detailed description of the invention

The present invention concerns the manufacture of an aqueous detergent composition. Said aqueous detergent composition comprises water, a nonionic surfactant, an anionic surfactant and a perfume. The compositions according to the present invention can be formulated in a variety of different embodiments, especially household cleaners.

As a first essential ingredient, the compositions manufactured according to the present invention comprise a nonionic surfactant according to the formula



- R_1 is a C_{1-25} alkyl or alkenyl chain, preferably C_{10-15} , preferably alkyl;
- R_2 is a C_{2-4} aliphatic hydrocarbon chain, preferably C_2 ;
- R_3 is a methyl or ethyl monosubstituted C_2-C_4 aliphatic hydrocarbon chain, preferably a methyl substituted ethylene;
- R_4 is a C_{1-25} alkyl or alkenyl carboxyl chain, or H, preferably H;
- n is an integer of from 1 to 10, preferably 1 to 5;
- m is an integer of from 0 to 20; preferably 0 to 10, most preferably 0 to 3; or mixtures thereof.

It is to be understood that, in the chemical formula above, R_2O and R_3O groups may appear in any sequence in the molecule; also, when n and m are greater than 1, different R_2O and R_3O groups may appear in a same molecule. Commercially available nonionic surfactant compounds are described for instance in the European Patent Applications EP-A-0 518 401 or EP-A-0 517 996. The compositions manufactured according to the present invention comprise from 0.1% to 30% by weight of the total composition of said nonionic surfactant or mixtures thereof, preferably from 0.5% to 10%.

As a second essential ingredient, the compositions manufactured according to the present invention further comprise an anionic surfactant or mixtures thereof. Suitable anionic surfactants for use herein have been extensively described in the art and include alkyl benzene sulphonates and alkyl sulphates. Alkyl sulphate surfactants for use herein can be obtained from natural source, e.g coconut. Accordingly, the compositions manufactured according to the present invention preferably comprise from 0.5% to 40% by weight of the total composition of said anionic surfactant or mixtures thereof, preferably from 0.5% to 10%. Particularly preferred herein is sodium coconut alkyl sulfate.

The compositions manufactured according to the present invention may further comprise additional surfactants such as described in the art including cationic and zwitterionic surfactants.

As a third essential ingredient, the compositions manufactured according to the present invention further comprise from 0.03% to 5% by weight of the total composition of a perfume, preferably from 0.03% to 3%.

The compositions manufactured according to the present invention comprise from 60% to 99% by weight of the total composition of water, preferably from 80% to 95%.

The compositions manufactured according to the present invention may also comprise additional conventional ingredients such as solvents, hydrotropes, chelating agents, thickeners, fragrance and whitening agents, provided all these ingredients are compatible with the compositions. Said ingredients are not critical for the clarity of the compositions of the present invention.

In a preferred embodiment the compositions manufactured according to the present invention also

comprise from 1% to 15% by weight of the total composition of a bleach, preferably from 2% to 10%. Preferred bleach for use herein is hydrogen peroxide.

5 The compositions manufactured according to the present invention are preferably acidic and have a pH of from 2 to 6, preferably 3 to 5, most preferably 4. Such pH range can be attained by the addition of appropriate acidifiers such as organic or inorganic acids, acidic salts which buffer pH
10 to an acid value. Examples of suitable acidifiers are sulfuric acid, phosphoric acid although it is somewhat undesirable from an environmental viewpoint, hydrochloric acid, phosphonic acid, citric acid, acetic acid, tartaric acid, maleic acid and the like.

15 In a further preferred embodiment the compositions manufactured according to the present invention further comprise butyl hydroxy toluene as an antioxydant and a H₂O₂ stabilizer. Said compositions comprise from 0.01% to 1% by
20 weight of the total composition of butyl hydroxy toluene, preferably from 0.01% to 0.8%.

 The process according to the present invention comprises the following steps.

25 In one step the nonionic surfactant is mixed in water so as to obtain a composition, hereinafter composition A.

 In an other step the perfume is dissolve in the anionic
30 surfactant so as to obtain a composition, hereinafter composition B.

 The order in which compositions A and B are made is not critical for the present invention.

35 In a further step of the process according to the present invention, said composition B is mixed in said composition A so as to obtain composition C. In said st p, the composition of

the present invention becomes clear and transparent when said compositions A and B are mixed together.

In a preferred embodiment where the compositions manufactured according to the present invention further comprise a bleach, said bleach is added on top of the composition C, in the final step of the process.

In a further preferred embodiment where the compositions manufactured according to the present invention further comprise butyl hydroxy toluene, said butyl hydroxy toluene is dissolved in the perfume before the step where said perfume is dissolved in said anionic surfactant so as to obtain a composition B.

In a further preferred embodiment where said aqueous bleaching composition further comprise acidifiers, it is preferred to include an additional step in the process of the present invention, where said acidifiers are added to the composition C before the final addition of the bleaching compounds. The acidifiers can be added pure or pre-diluted.

Example

The following compositions were made by the listed ingredients in the listed proportion according to the process of the invention as described hereinafter.

Compositions	1	2	3	4	5	6
-----	----	----	----	----	----	----
	Weight %					
H ₂ O ₂	6	8	8	6	5	5
alkyl sulphate	3.5	4	1	1	3	1
Citric acid	6	0	4	6	4	4
Polyethoxypropoxy						
alcohol	1.5	4	1	1	1	1
perfume	0.5	0.1	0.1	0.3	0.5	0.1

7

Butyl hydroxy toluene	0.03	0.03	0.03			0.03
NH ₃ up to pH	4				4	
NaOH up to pH			3	4		3.5
H ₂ SO ₄ up to pH		4				
5 Water & minorsup to 100%.....					

10 In one step of the process according to the present invention the polyethoxypropoxy alcohol is mixed in water. The composition obtained is named A.

15 In another step, the butyl hydroxy toluene is dissolved in the perfume. After that, the perfume is dissolved in the anionic surfactant. The resulting composition is named B.

Thereafter, in a further step of the process according to the present invention, composition B is mixed in composition A.

The pH is adjusted.

20 And finally, hydrogen peroxide is added as a final step in the process, on top of the remainder of the composition.

25 The present compositions prepared in accordance with the specific mixing order of the present invention is clear and transparent.

WHAT IS CLAIMED IS:

1. A process for the manufacture of an aqueous detergent composition comprising a nonionic surfactant, an anionic surfactant and a perfume characterized in that said process includes the steps of:

- mixing said non-ionic surfactant in said water (composition A);
- dissolving said perfume in said anionic surfactant (composition B);
- thereafter mixing said composition B in said composition A (composition C);

2. A process for the manufacture of an aqueous detergent composition according to claim 1, wherein said nonionic surfactant is according to the formula

$$R_1-O-[(R_2O)_n(R_3O)_m]-R_4, \text{ wherein :}$$

- R_1 is a C_{1-25} alkyl or alkenyl chain;
- R_2 is a C_{2-4} aliphatic hydrocarbon chain;
- R_3 is a methyl or ethyl monosubstituted C_2-C_4 aliphatic hydrocarbon chain;
- R_4 is a C_{1-25} alkyl or alkenyl or carboxyl chain, or H;
- n is an integer of from 1 to 10;
- m is an integer of from 0 to 20; or mixtures thereof.

3. A process for the manufacture of an aqueous detergent composition according to the preceding claims, wherein said composition comprises from 0.1% to 30% by weight of the total composition of said nonionic surfactant.

4. A process for the manufacture of an aqueous detergent composition according to the preceding claims, wherein said composition comprises from 0.5% to 40% by weight of the total composition of said anionic surfactant.

5. A process for the manufacture of an aqueous detergent composition according to the preceding claims, wherein said composition comprises from 0.03% to 5% by weight of the total composition of said perfume.

5

6. A process for the manufacture of an aqueous detergent composition according to the preceding claims, wherein said composition further comprises a bleaching compound, and wherein said bleaching compound is added as a final step to said composition C.

10

7. A process according to claim 6 for the manufacture of an aqueous detergent composition, wherein said composition comprises as said bleaching compound from 1% to 15% by weight of the total composition of hydrogen peroxide.

15

8. A process for the manufacture of an aqueous detergent composition according to the preceding claims, wherein said composition further comprises butyl hydroxy toluene, and wherein said butyl hydroxy toluene is dissolved in the perfume before the step where said perfume is dissolved in said anionic surfactant.

20

9. A process according to claim 8 for the manufacture of an aqueous detergent composition, wherein said composition comprises from 0.01% to 1% by weight of the total composition of said butyl hydroxy toluene.

25

10. A process for the manufacture of an aqueous detergent composition according to the preceding claims, wherein said composition comprises from 60% to 99% by weight of the total composition of water, preferably from 80% to 95%.

30

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US94/04329

A. CLASSIFICATION OF SUBJECT MATTER

IPC(5) : C11D 1/02, 1/72, 3/50

US CL : 252/174.11, 173, 174, 21, Dig 14

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 252/174.11, 173, 174, 21, Dig 14

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

APS

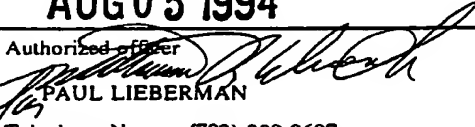
Search terms: anionic, nonionic, perfume, detergent or clean?, water or aqueous

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US, A 4,608,189 (Koch et al.) 26 August 1986 See col.2, lines30-40, col.3, lines40-54.	1-2
Y	EP, A, 0 517 996 (SCALIA ET AL.) 16 DECEMBER 1992; see col. 1, lines 40-45./	1-2
Y	US, A 4,787,998 (Rennie et al.) 29 November 1988. See col. 2, lines 21-27, and lines 5-15.	1-2
Y	EP, A, 0 518 401 (Carrie et al.) 16 December 1992. See Col. 3, lines 30-40.	2

☐ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

* Special categories of cited documents:	*T later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
A document defining the general state of the art which is not considered to be of particular relevance	*X* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
E earlier document published on or after the international filing date	*Y* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
L document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	*G* document member of the same patent family
O document referring to an oral disclosure, use, exhibition or other means	
P document published prior to the international filing date but later than the priority date claimed	

Date of the actual completion of the international search 07 JULY 1994	Date of mailing of the international search report AUG 05 1994
Name and mailing address of the ISA/US Commissioner of Patents and Trademarks Box PCT Washington, D.C. 20231 Facsimile No. (703) 305-3230	Authorized officer  PAUL LIEBERMAN Telephone No. (703) 308-0687

INTERNATIONAL SEARCH REPORT

International application No.

PCT/US94/04329

Box I Observations where certain claims were found unsearchable (Continuation of item 1 of first sheet)

This international report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claims Nos.:
because they relate to subject matter not required to be searched by this Authority, namely:

2. ☐ Claims Nos.:
because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:

3. ☒ Claims Nos.: 3-10
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

Box II Observations where unity of invention is lacking (Continuation of item 2 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

1. ☐ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims.
2. ☐ As all searchable claims could be searched without effort justifying an additional fee, this Authority did not invite payment of any additional fee.
3. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims for which fees were paid, specifically claims Nos.:

4. ☐ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

Remark on Protest

- ☐ The additional search fees were accompanied by the applicant's protest.
- ☐ No protest accompanied the payment of additional search fees.

THIS PAGE BLANK (USPTO)